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**WASHINGTON STATE UNIVERSITY
COLLEGE OF ENGINEERING
RESEARCH DIVISION**

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Bulletin 310

WATER RECLAMATION BY REVERSE OSMOSIS

by

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ABSTRACT

The reverse osmosis process, which employs a cellulose acetate membrane, offers a means of producing water low in inorganic and organic substances from single solute aqueous systems, multi-solute aqueous systems, and secondary-treated sewage effluents. Compounds when in an aqueous system having significantly higher vapor pressure than water may appear in significant quantities in the reclaimed water. This process can also serve as a means of concentrating weak waste water to a point where the waste water can be more economically treated.

The permeation of 55 chemical species singly in an aqueous solution and dispersion, and/or in multi-component systems were investigated. The degree of permeation of radioactive nuclei and of some enteric microorganisms are also reported on.

INTRODUCTION

Water is a prime commodity of any nation. With an unlimited supply of a usable quality water, the economic growth of a region can flourish. Without it, economic growth cannot take place.

A good supply of water is particularly vital for the growth of urban areas. It has been predicted that within the next two decades at least two-thirds of the population of the United States will reside in metropolitan centers with much of the nation's industry being located in or adjacent to these areas. This continued increase in population and industry will make the procurement and supply of water an important national concern.

However, many bodies of surface water now used as raw water supplies also serve as recipients of waste water from industries, municipalities, and croplands. Although these raw water supplies have not noticeably decreased in volume, their use as waste water receivers has increased, resulting in many cases in quality degradation. Treated and untreated waste waters are discharged into the streams and/or groundwater strata. The next user down-gradient reuses in part the discharged waste water. This process is repeated many times until the water reaches the ocean. This frequently repeated reuse of the water alters the quality to an extent where it can no longer meet the health, process, and aesthetic standards of the down-gradient users.

It is not that these surface waters are receiving poorer quality treated waste water effluent, but rather that the effluent is being discharged in larger and larger amounts. It has long been recognized that conventional secondary treatment can remove no more than 95 per cent of the decomposable

organic matter from the waste water. In addition, the treated effluent may contain up to ten times as much non-degradable organic matter as it does degradable organic material. A number of these non-degradable or partially-degradable organic compounds are suspected to have cumulative toxic effects, either singly or in combination.

Also, conventional waste treatment may not remove any or only specific inorganic substances from the waste water. Thus in repeated reuse of this water, inorganic substances will tend to accumulate, resulting in a higher mineral content.

This increased concentration of organic and inorganic matter may have a secondary effect on the recipient body of water by stimulating the growth of objectionable aquatic microorganisms. In turn, these microorganisms may have a very pronounced effect on the quality of the water.

Since conventional water treatment methods do not remove all the soluble inorganic and organic matter from a raw water supply, what can communities do that are facing a decline in quality of their water supply, and/or increasing water demand? Desalination of sea water is feasible only if these cities are situated near the ocean. However, this is not feasible for inland urban areas. Therefore, what other alternative is there but the direct reuse of the communities' own waste water after some type of tertiary or advanced water treatment? The problem thus resolves itself into finding a method or methods that will economically reclaim fresh water from waste water.

One method which shows promise is reverse osmosis. The use of this procedure to desalinate seawater and brackish water has been studied by Breton and Reid (1) and Yuster, et al. (2), since the early nineteen fifties.

Since then a vast amount of work has been published on the many basic and applied aspects of this procedure (3).

The potentials of the reverse osmosis process led to an investigation by the Sanitary Engineering research staff at Washington State University to determine the applicability of reverse osmosis as: (a) an advanced waste treatment method, (b) a method of concentrating dilute waste streams, and (c) a method of producing a potable water from waste water.

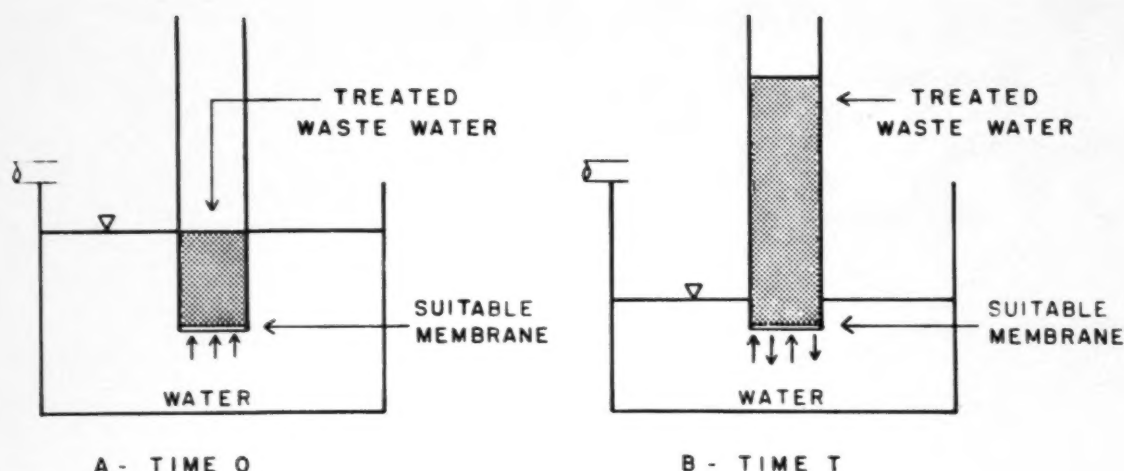
REVERSE OSMOSIS

The basic principles of the osmosis phenomena will be reviewed in this section so that reverse osmosis may be better understood.

When an aqueous solution is separated from water by a suitable membrane, the water molecules will permeate through the membrane into the solution. This process is known as osmosis. The permeation of the water molecules into the aqueous solution will result in a decrease in the original mass of the water with an increase in the mass of the aqueous solution.

Figure 1 shows what takes place when a tube containing waste water - and having one end of the tube sealed with an ideal semi-permeable membrane - is placed in a container of water. The level of the waste water in the tube and that of the water in the container are the same at time "zero". After a period of time, "t", it can be observed that the water will be at a lower level than at time "zero", while the level of the treated waste water in the tube will have risen.

FIGURE 1
The Process of Osmosis



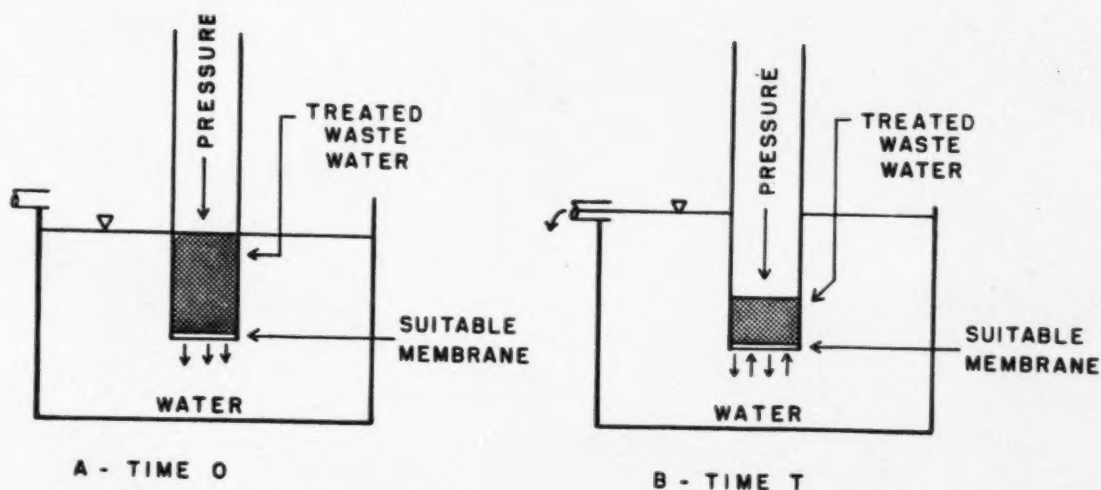
It appears that in the osmotic phenomena, a permeation of diffusion pressure exists which forces water molecules to diffuse through the membrane from a region of high concentration of water to that of a low concentration of water. As the treated waste water solution rises in the tube, a hydrostatic pressure develops in the tube. Development of the hydrostatic pressure increases the tendency of the water molecules in the treated water solution to permeate back into the water. Eventually a state is reached where the hydrostatic pressure equals the diffusion or permeation pressure. The pressure differential under the dynamic equilibrium state is known as the osmotic pressure.

If pressure equal to the osmotic pressure is applied to the waste water

tube, the level of both water and waste water will return to their original level or to the level of time "zero". This is shown in Figure 2. If a pressure in excess of the osmotic pressure is exerted on the waste water, water molecules will continue to diffuse out of the waste water into the water container. The flow from the treated waste water tube will continue until a new equilibrium is reached. In essence, this is what occurs during the reverse osmosis process.

FIGURE 2

The Process of Reverse Osmosis



OSMOTIC MEMBRANE

The most vital part of the reverse osmosis system is the membrane. An ideal membrane is one which would allow only water molecules to pass through its structure. This type of membrane is termed semi-permeable or differentially permeable to water.

There are a number of substances which have been used in fabricating semi-permeable membrane films. A modified cellulose acetate material (11) (6) has proven to be most satisfactory in demineralization studies. Other types of membrane films being investigated are carbon, synthetic polymers, inorganic crystalline solids, and macromolecules occurring in the waste water (4). In the investigation reported herein, modified cellulose acetate membranes were used.

PERMEATION MECHANISMS

Cellulose acetate films are classified as neutral membranes in that cellulose acetate itself does not possess strong active ion exchange properties.

There are three principal theories regarding the permeation through an ideal membrane. These theories are the membrane solution theory, the vapor pressure theory, and the molecular sieve or pore theory. The latter two appear to explain the transport of water and other species through a porous cellulose acetate membrane.

Certain membranes permit only the solvent to diffuse from one surface through its bulk to the opposite side of the film. This phenomenon is the basis of the membrane solution theory. In the case of an aqueous solution, water molecules would be the only species to permeate through the membrane film.

In the vapor pressure theory, the membrane acts as a pseudo-vapor distillation apparatus. Water in the form of water vapor permeates through the membrane micropores. Any other species more volatile and spatially equal to or smaller than the water vapor species will also be transported through

the membrane. Since vapor pressure is a critical factor, a pressure must be applied to a solution to overcome the lowered vapor pressure of water in solution compared to pure water. The lower the mole fraction of water in the solution the lower the vapor pressure of the water in solution.

The molecular sieve or pore theory assumes that the membrane acts as a filter permitting the passage of ions and spatially small molecules while preventing the permeation of spatially large molecules and ionic complexes. Reid and Breton (5) extended this theory, contending that the transfer of water and ions through a cellulose acetate membrane is dependent on two factors: One, those species that can associate with the membrane through hydrogen bonding actually combine with the membrane and are transported through the film by an alignment-type diffusion. Two, those species which are not capable of hydrogen bonding with the membrane are transported through by the hole or pore-type diffusion method.

Sourirajan (6) theorized that transport by the molecular sieve or pore theory is a surface phenomenon. A thin multi-molecular layer of deionized water exists at the interface between the membrane surface and aqueous solution. This multilayer of water molecules is continually being formed as the layer is removed. The thickness of the multi-layer is dependent on the concentration of the species in the solution. The deionized water flows through the pores of an appropriate size. A pressure is required to sustain a continuous flow of water in the liquid state through the capillary pores of the membrane.

Though cellulose acetate membranes are classified as neutral, it is possible for the film to obtain an induced charge. Thau, et al. (7) found the existence

of selective adsorption of one type of ion on the membrane surface causing a neutral type of membrane to become a pseudo-ion exchange type. The work done by Thau, et al. (7) indicates that this occurs with cellulose acetate films.

A complete explanation is not available of the permeation mechanisms involved in the reverse osmosis process where a cellulose acetate film is used as the membrane material. Observations are available which indicate the boundary surface-molecular sieve or pore theory is applicable where volatile species are not involved, while the vapor pressure theory seems credible where the presence of species more volatile than water exists. An induced charge on the membrane surface is also believed to occur after the membrane has been in use for a period of time.

MEMBRANE STRUCTURE

Riley, et al. (8) (9), used electron microscopy to study the structure of the cellulose acetate membrane. From the micrographs they obtained, it was observed that the membrane consists of a dense surface layer devoid of structural characteristics, and a porous, ill-defined substructure layer. The dense surface layer was about 1/40 of the total thickness of the membrane and possessed capillary pores. The spongy substructure layer had a pore size on the order of 0.1μ . When pressure was applied to the dense surface layer, its structure remained unchanged while a degree of compaction of the spongy subsurface was noted. Their studies revealed that the water transmission rate of the different cellulose acetate films differed approximately by the inverse thickness of the dense surface layer.

It was observed that the dense surface layer must be exposed to the

aqueous solution in order for the membrane to be semi-permeable. If the spongy subsurface side is exposed to the solution the transport of all spatially small species occurs.

TERMINOLOGY

There are a number of terms used repeatedly in describing and/or evaluating the efficiency and operation of the reverse osmosis process. These are as follows:

Feed water - An aqueous solution from which the water is to be removed.

Product water - The water which has permeated through the membrane.

Raffinate or concentrated feed - The aqueous solution which has had part of its water content removed.

Cell flow rate - The rate of flow through the reverse osmosis cell, expressed in terms of gallons per day or cubic meters per day.

Product flow rate - The rate of flow of product water from the cell, expressed in the same terms as the cell flow rate.

Membrane flux - The quantity of water permeating through a unit membrane surface during a time interval. This term is expressed either as gallons of product water per square foot of membrane surface per day or cubic meters of product water per square meter of membrane surface per day.

Pressure - The hydrostatic pressure applied to the feed water. It is expressed in terms of pounds per square inch or atmospheres.

Cycles - The number of times the raffinate or concentrated feed is recirculated through the reverse osmosis cell.

Feed-product water ratio - The ratio of the quantity of feed used in a unit time period to the quantity of product water produced in the same unit time period.

$$R_q = \frac{\text{Volume of feed per day}}{\text{Volume of product water per day}}$$

Reduction factor - This term is used to measure process efficiency.

$$R = \frac{\text{Concentration of the species in the feed}}{\text{Concentration of the species in the product}}$$

Per cent reduction =

$$\frac{\text{Conc. of the species in feed} - \text{Conc. of species in product}}{\text{Conc. of the species in feed}} \times 100$$

The per cent reduction may also be expressed as the mole percentage of solute removed.

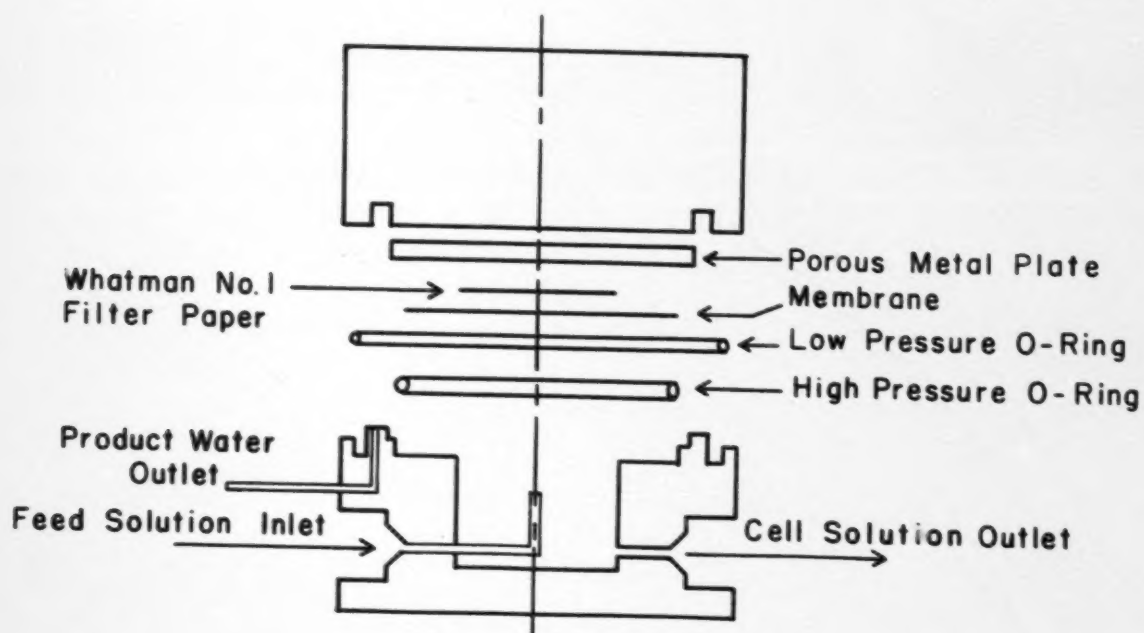
PROCESS OPERATION

A 2.75 inch (7.00 cm) diameter laboratory scale reverse osmosis cell was used in the study reported on in this bulletin. The basic design is similar to that used by Loeb (10). Figure 3 is an enlarged view of the cell. (Next page.) The cell was fabricated from stainless steel except for the neoprene "O"-rings. The two sections were clamped together in a modified screw press when in use.

The cell operates in the following manner: The feed solution enters the cell through the feed solution inlet into a feed chamber. From the chamber the feed solution flows past the membrane. Water molecules permeate through the membrane across the filter paper and porous plate backing into a trough on the top section of the cell. The water is conveyed along the trough to the product

water outlet opening, thence out through the product water tube. The wasted feed or raffinate leaves the cell through the cell solution outlet.

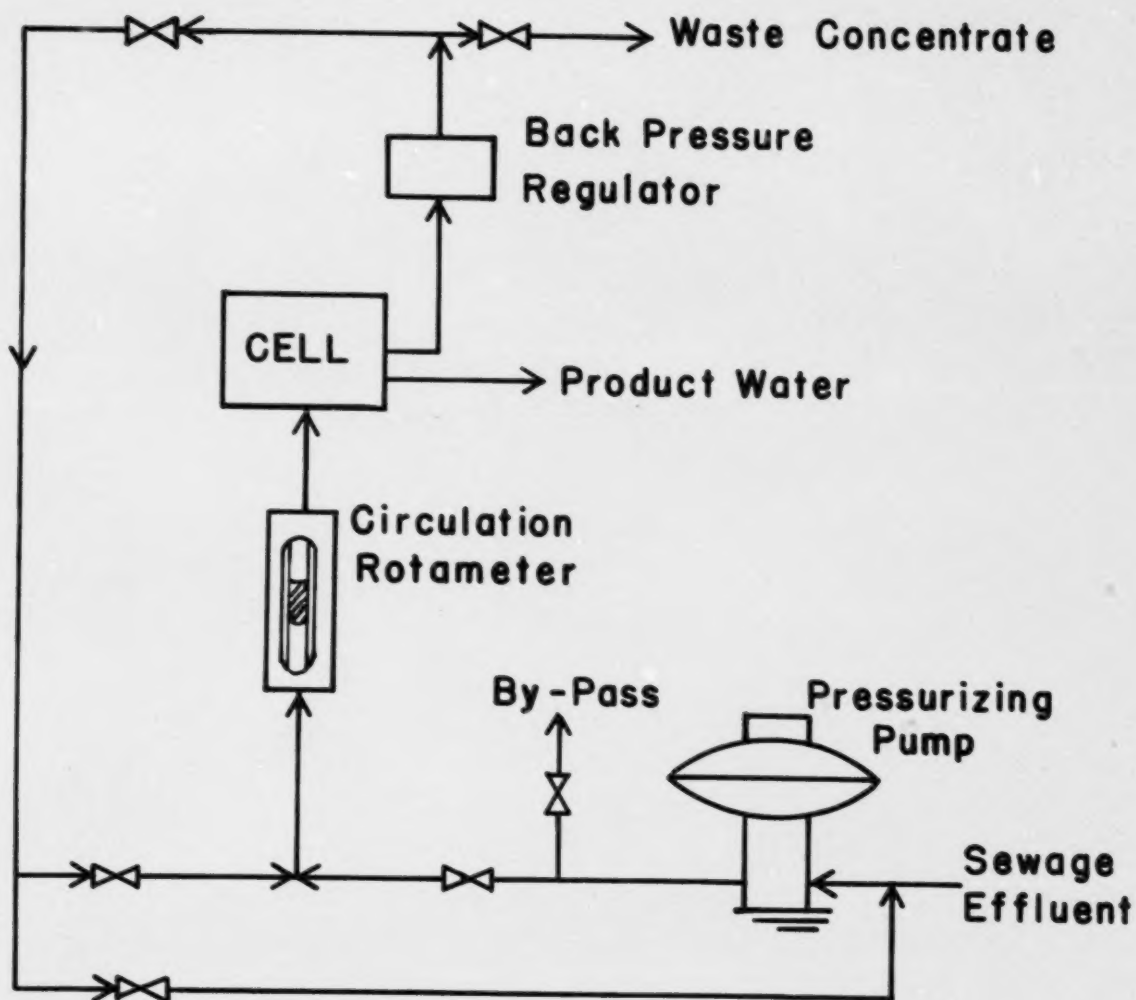
FIGURE 3
Laboratory Scale Reverse Osmosis Cell



The feed is pumped into the high-pressure reverse osmosis cell via a rotameter by a pressurizing pump. Two streams leave the cell, the product water, and the raffinate or concentrated feed. The product water is collected. The raffinate flows through a back pressure regulator and is either wasted or recycled. Valves are placed in appropriate positions to facilitate recycling, draining, or cleaning the system.

FIGURE 4

Flow Diagram of a Reverse Osmosis System



MEMBRANE MODIFICATION

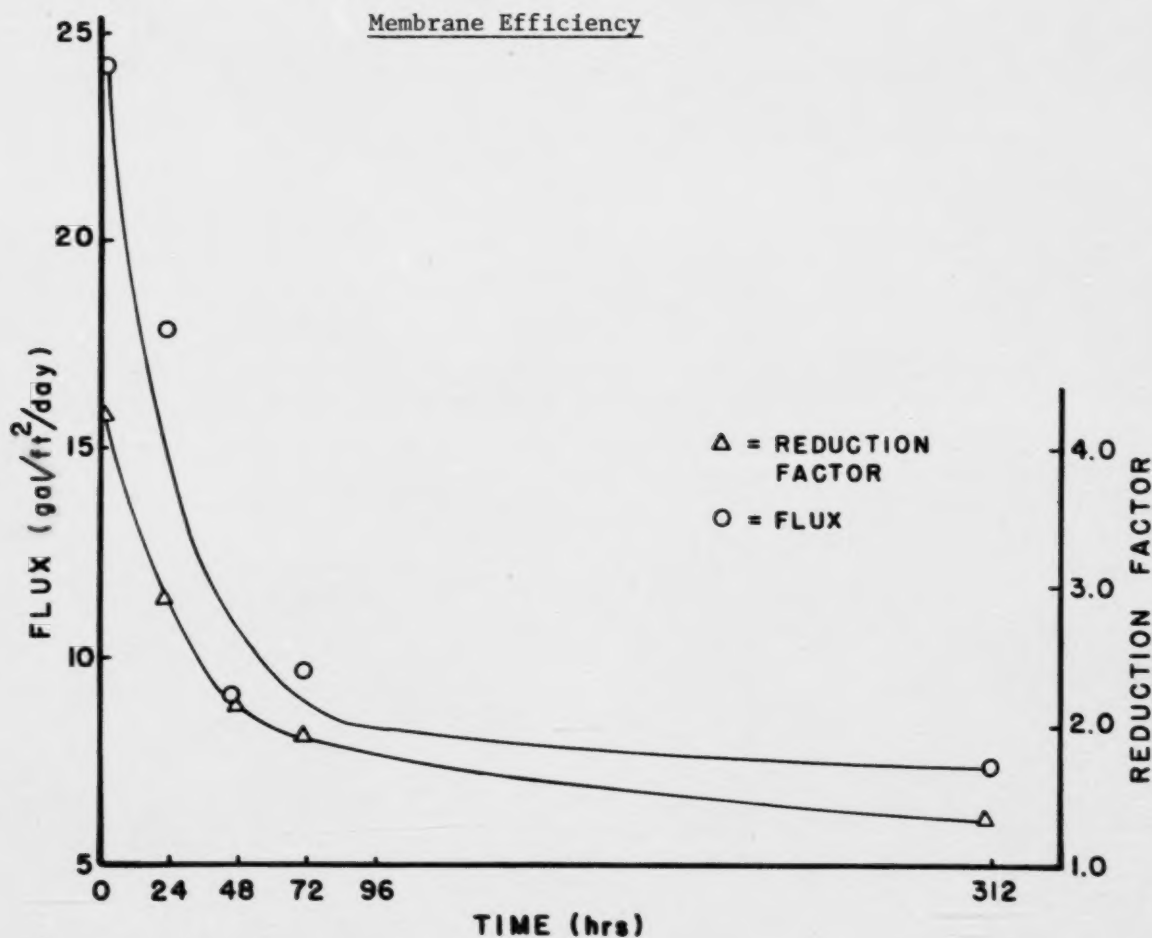
An investigation was conducted to determine the transfer properties of a number of commercially available cellulose acetate membranes. Loeb (11) and Sourirajan (6) found that shrinking the membrane resulted in higher reduction factors. Shrinking is thought to reduce the pore size, thereby limiting the

size of the species permeating through the membrane. Table I (at the end of the bulletin) shows the effect of shrinking on membrane efficiency and flux.

A Schleicher and Schuell Type B-20 membrane gave the greatest reduction factor. However, the flux was of such a magnitude as to make it impractical to use. Type B-18 was used as it gave a fairly good reduction factor at a reasonable flux rate.

Not only is the membrane the heart of the reverse osmosis system, it is its weakest point. With increased membrane usage, a reduction in the flux and reduction factor occurs. Figure 5 illustrates this effect graphically.

FIGURE 5



In determining membrane efficiency, a solution containing a mixture of inorganic ions - having a conductance of 595 micromhos at 25°C. - was used. A pressure of 102 atmospheres was applied to the reverse osmosis system.

Other investigators, among them Loeb (12) and Sourirajan (13), have reported a decrease in efficiency and flux with increasing membrane usage. The magnitude of the loss in flux and efficiency will vary with the configuration of the membrane (tubular or disk shaped), the method of membrane fabrication, the composition of the membrane, and the composition and concentration of specific chemical species in the feed solution.

EXPERIMENTAL PLAN

It is known that sewage and many other types of waste water are heterogeneous mixtures. The kinds and concentration of specific chemical species and microorganisms present in the treated sewage effluent of one community may not be the same as that of another community. This is also true for treated waste water effluents from particular industrial processes. In addition, the chemical composition of the treated sewage or industrial waste water effluent may change during the day or week.

In this investigation, a study was conducted to determine the permeation of specific chemical species, rather than solely investigating the permeation of those substances found in the treated sewage of a few communities or of an industry. The species investigated are those found in abundance in most treated waste water effluent. Those species known or suspected to be toxic were also investigated, although they are not often found in waste water.

Initially, an aqueous solution or dispersion containing only one chemical

species was investigated to determine its ability to permeate through the membrane. Aqueous solutions or dispersions of each species at more than one concentration also were studied.

The second stage of the study consisted of investigating aqueous solutions or dispersions of a mixture of chemical species to determine the presence of synergistic or antagonistic action of one specific chemical species toward the permeation of another species. The final stage determined the capability of the membrane to prevent the permeation of specific chemical species in treated effluents or such effluents spiked with specific chemical species.

RESULTS

The data presented in Tables II, III, VII, VIII and IX are an average of five replicate runs for each species reported. The apparatus was operated at room temperature (approximately 25°C). Schleicher and Schuell Type B-18 membranes immersed at 82°C for 30 minutes were used throughout. A pressure differential of 100 atmospheres was applied to the feed solution.

Inorganic Compounds

Table II is a summary of the data obtained for feeds containing one specific inorganic species in solution.

A number of generalities can be concluded from the data presented in Table II. A decrease in the concentration of all chemical species, except for nitrates, fluorides and borates, resulted in either an increase in the percentage reduction or the percentage remained the same. However, caution must be used in comparing the percentage reductions of two or more particular

species at a particular concentration when the concentration is expressed on a mass per volume basis. A better comparison probably can be made on a mole fraction or molality basis. However, it must be remembered that ions most often do not exist as such, but rather in a hydrated or complexed form.

The data reveals that an order of ionic inability to permeate through a porous cellulose acetate membrane does exist. For the cationic ions it is as follows: $Al^{+3} > Fe^{+3} > Ca^{+2} > Na > NH_4-N > K > Cd^{+2} > Mg > Ca^{+2}$. A closer look at the cationic ions reveals that the trivalent ions can react like a Lewis acid and form hydroxy complexes. This is also true to a more limited extent with copper. Most cationic ions do form hydrates. If the spatial size of the ion is a factor in its ability to permeate through the membrane, then the hydration of ion data reported by Gluerkauf (14) must be considered. The spatial size of the ion complex or hydrated ion and the degree of hydrogen bonding also affect the ability of the chemical species to permeate through the membrane. The pH of the solution may be an important factor as the hydrogen concentration may dictate in what form the multi-valent cations will exist.

The data obtained by Sourirajan (13), and by Loeb and Manjikian (15), are in close agreement with the percentage reduction obtained in the study being reported on. Sourirajan contended that the percentage reduction follows the same sequence as the lyotropic series. The data reported in Table III is not in complete agreement with this assumption. The deviation from the sequence lies with the divalent cations.

An order of permeation of anionic ions through the membrane can also be formulated. The sequence is as follows: $SO_4 > CrO_2 > SiO_2 > Cl > PO_4 > F = CN > NO_2 > NO_3 > B_4O_7$. The sequence is in agreement with the limited data

reported by Sourirajan (13). Again, Gluerkauf's (14) data can be used in rationalizing the reason for the permeation order.

The data presented in Table II - the percentage of reduction of specific species solely in aqueous solution or dispersion - shows that the reverse osmosis process has merit in producing a water having a significantly low ionic concentration.

The permeation of each inorganic anion in a mixture was compared to each anion if present singly in solution to reveal synergistic or antagonistic effects on permeation. A mixture of the sodium salts of three anions was prepared. Table III is a summary of the data obtained. The difference in the percentage reduction of ions in a mixture as compared to the ion solely in solution is within the range of experimental error. Antagonistic or synergistic effects were not found.

Investigations were carried out to determine the permeation of the major inorganic constituents of secondary-treated sewage effluent. Two types of biologically-treated effluents were used. Table IV contains the permeation data obtained using trickling filter effluent as feed while Table V shows the data obtained using activated sludge effluent. A comparison was made of the percentage reduction using feeds consisting of trickling filter effluents, activated sludge, treated effluent, and aqueous solutions where the ion in question was the only solute. The comparisons are shown in Table VI.

In comparing the percentage reduction in using different feeds, the reverse osmosis process does an excellent job in producing a feed water low in inorganic substances. An exception to the last statement is the removal of nitrates from

a feed of secondary biologically treated sewage effluent. The pH of the product water was slightly lower than the feed. This was probably due to the buffering capacity of the product.

In most cases, the percentage of reduction of specific ions in the product water, when sewage effluent was used as feed, was greater than the specific ion singly in aqueous solution, either based on only the concentration of that ion in solution or based on the total ions in solution. The greater reduction in the percentage reduction of specific cations is probably due to the greater tendency for these ions to become complexed with organic material. The pH of the sewage effluent feed was conducive for the formation of hydroxy complexes of the trivalent cations. This was not apparent with the ferric ion because the ferric ion was in low concentration, near the minimum detectable limit. Change was not very apparent in the percentage reduction of anionic substances in the product water using sewage effluent as a feed compared to the ion singly in aqueous solution. The slight changes in percentage reduction was probably due to the fact the hydration was the only mode of reaction. Hydration would occur with the anion regardless of the type of feed solution.

It can be concluded that extreme discretion must be used in projecting the percentage reduction of any specific inorganic substance in waste water effluent feeds from the data obtained for that substance solely in aqueous solution.

Radioactive Species

The reverse osmosis process was used as one of the means of concentrating the radionuclei in a study by Merana (16) on concentrating chromium-51 liquid wastes. Radioactive chromate ion was produced from chromium-51 chloride by oxidation.

Operational parameters such as flow rate and applied pressure were investigated in this study to find those conditions which would give the maximum concentration of chromium-51 in the raffinate. An applied pressure of 1000 psig and a flow rate of 38 gallons per day gave the maximum degree of concentration of the radioactive nuclei. The permeation data of the chromium nuclei as the chromate ion in dilute solution through a porous cellulose acetate membrane was compared with that of an aqueous solution of the non-radioactive form of chromium. Table VII summarizes the results.

It was found that the percentage reduction in the product water using chromium-51 as the chromate ion was the same (within experimental error) as the reduction obtained with the stable chromate ion. It can be concluded that the percentage reduction of an radioactive species is the same as the stable species.

Permeation of Organic Compounds

The same scheme of study used for the permeation of inorganic ions was utilized to investigate the permeation of organic chemical species across a cellulose acetate membrane. Initially, organic compounds were studied solely in aqueous solution or in dispersion. Then these compounds were studied in mixtures, and finally their permeating ability investigated when found in treated sewage effluent or added to the effluent.

Table VIII is a compilation of the permeation data of organic species in aqueous solution or in dispersion. The operational conditions of the cell were as follows: temperature about 25°C., pressure differential 100 atmospheres, and Schleicher and Schuell Type B-18 membrane immersed in water at 82°C. for 30 minutes.

The data in Table VIII reveal that a general statement cannot be made

concerning the permeation of organic compounds through a cellulose acetate membrane. However, some generalities can be concluded by classifying the organic compounds in groups based on their physical state in solution or dispersion, the possibility of existence in more than one chemical form, and on their volatility in an aqueous medium.

Certain species will form colloidal particles, aggregates, micelles, or macromolecules in an aqueous medium. These species are spatially large groups. Detergents (ABS and LAS), soaps (Sodium oleate), motor oil, DDT, TDE (DDD), proteins, starch, cellulose, humic acids, and tannins are found primarily in this form. Not only does DDT exist in the aggregate form but it will exist in true solution in concentrations below one microgram per liter, according to Acree, et al. (17). Other insecticides are known to be much more soluble in water than DDT and TDE. There is some question concerning the predominant form of lindane and BHC existing in water below a concentration of 100 micrograms per liter.

Reductions in the 80-99 per cent range were obtained for those chemical species existing primarily in the colloidal, aggregate, micelle, or macromolecular form. If a chemical species exists both as an aggregate in dispersion or as a discrete molecule in true solution where the vapor pressure of the discrete molecule is appreciably greater than that of water, the reduction was found to be in the 50 to 80 per cent range.

Where discrete molecules more volatile than water are present in solution or suspension, as in the case of p-chloro nitrobenzene and the lower molecular weight esters, the reductions were in the 14 to 40 per cent range. Higher percentage reductions were obtained for the higher molecular weight and less volatile isopropyl ester of 2,4-D.

A very limited amount of data appears in the literature concerning the permeation of specific organic compounds across a cellulose acetate membrane. Ironside and Sourirajan (18) reported that a greater than 99 per cent reduction in detergents was obtained when using a feed solution containing 512 mg/l of ABS or a 370 mg/l solution of sodium dioctyl sulfo succinate. In a study by Sourirajan and Sirianni (19), it was found that the percentage removed by the membrane for several of the polyethylated nonionic surfacants varied from 69 to 90 per cent using a very dense Schleicher and Schuell membrane. A greater reduction (90-99 per cent) was obtained using their laboratory-prepared membrane.

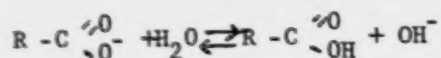
An acidic brown lignin solution was used as a feed by Ironside and Sourirajan (18). They were able to obtain a reduction greater than 99 per cent. Sourirajan (6) investigated the permeation of low molecular weight alcohols, carbonyl compound, and mono and disaccharides in aqueous solution. Of the same organic compounds investigated by Sourirajan and those found in Table VIII, a greater percentage reduction is reported in Table VIII. However, the concentration of acetone in the feed used by Sourirajan was in the percentage range found in this study.

In the study reported herein, the effect was investigated of organic species on the permeation of a specific organic species in solution or dispersion. The permeation data of specific chemical species in a multiple component aqueous solution or dispersion are presented in Table IX. The data in Table IX show a marked increase in the reduction factor of methyl formate in an aqueous solution containing three other low molecular weight esters than when methyl formate was present solely in solution. This information, in addition to some of the results

obtained in the phenol-chlorophenol study, indicates that the vapor pressure theory of transport through a porous cellulose acetate membrane has some credence.

The results of the investigation of mixtures consisting of 2,4-D isopropyl ester, p-chloro nitrobenzene, phenol, chlorophenol, and ethyl acetate shows that antagonistic as well as synergistic effects are present at the membrane surface. This may be due to a change in the mole fraction of the species in a mixture compared to the species solely in solution. A significant decrease in the percentage reduction of 2,4-D isopropyl ester and chlorophenol was observed. The magnitude of change of the other constituents are considered to be within the range of experimental error. However, these minor changes in percentage reduction were in a direction of a decrease, with the exception of ethyl acetate. From the results obtained it can be concluded that synergistic effects are assisting these volatile species to permeate through the membrane.

There are a number of organic compounds that when dissolved in water will enter in true solution and form ions. Some of these also have the ability to be present in more than one form, e.g.,



The direction of the reaction will depend on a number of factors, among which are the pH of the solution and removal of one of the reactants or products. In the case of the monocarboxylate compounds, the acid form is much more volatile than the ionic form. In addition, the hydration property is different for each. The degree of hydration is extremely important in the permeation of the amino acids. The degree of permeation of one form of the amino acid over another form may be the reason for the data discrepancy for the amino acid mixture. Therefore, it is not surprising that the percentage of permeation of glycine was in the same range as the lower molecular monocarboxylates.

The percentage reductions of specific monocarboxylates were in the same range as that of BHC and lindane. Though the aggregate-discrete molecule relationship does not exist with the monocarboxylates, ionized-nonionized species do exist. One member of the relationship does have a higher vapor pressure than the other. Comparing the percentage reduction of the amino acids and the monocarboxylates with the other discrete organic molecules in solution indicates that a mechanism other than one based on the vapor pressure theory must occur.

The permeation through a porous cellulose acetate membrane of a number of organic compounds found in sewage effluents was investigated. The sewage effluent was centrifuged, then sterilized prior to being used as a feed. Where the specific species were absent in the effluent, the effluent was "spiked" with the material. The data from this investigation are shown in Table X.

A comparison of the percentage of reduction of specific species in sewage effluent, aqueous multi-component systems, and singly in solution or dispersion is shown in Table XI. The percentage reduction of ABS in trickling filter effluent was within the range obtained when the detergent was solely in aqueous solution. The percentage reduction of the anthrone-reactive material (presumed to be carbohydrate) varied depending upon the type of effluent used. The percentage reduction of the anthrone-reactive material using trickling filter effluent as the feed was similar to that of cellulose. However, when settled activated sludge effluent was used, the percentage of reduction was in the range of the results obtained using starch.

The most significant change in the percentage reduction of organic species occurred with a sample of sterilized activated sludge-treated effluent which

had been "spiked" with a mixture of the isopropyl ester of 2,4-D, p-chloro nitrobenzene, phenol, chlorophenol, DDT, ethyl acetate, and acetone. The percentage reduction of each species in the effluent, with the exception of ethyl acetate, was the same as or greater than that obtained in either an aqueous mixture or singly in solution. If the permeation data were known when the species was singly in solution, a close approximation of the percentage reduction of a specific species in sewage could be estimated.

The percentage reduction of glycine, using an activated sludge treated effluent feed "spiked" with amino acids, was significantly greater than when glycine was in aqueous solution in a multicomponent amino acid mixture. This may be due to a pH effect whereby glycine is far beyond its isoelectric point. A decrease in the percentage reduction of phenylalanine, using sewage effluent as a feed, was less than when these two amino acids were in solution. The change in percentage reduction may be due to the pH and buffering action of the activated treated effluent feed.

Microorganisms

Microbiological studies were conducted to determine the permeation through a porous cellulose acetate membrane of microorganisms found in sewage effluent. In carrying out the study the following conditions were used: cell temperature approximately 25°C., pressure 100 atmospheres, and flux range 15.0 to 19.2 gals/ft²/day. Prior to use in the study the membrane gave a 94.2 per cent reduction in sodium ion. The results obtained are shown in Table XII.

The data show that except for one run the microorganisms did not permeate through the membrane. The inability for the microorganisms to permeate through

could be anticipated if they are regarded as large macromolecules. The membrane serves as a fine filter and retains the microorganisms in the feed water or raffinate as the microorganisms are too large to travel through the pores of the membrane.

The product water was free of microorganisms except for the test when a feed containing 540 E. coli per ml was used. Their presence in the product water may have been due to an imperfection in the membrane or contaminate in the product water line. Bacteriophage of approximately the same shape and size as enteric viruses did not permeate through a porous cellulose acetate membrane.

Comparable results to those reported in Table XII were obtained by Ironside and Sourirajan (18). Sterile product water was found in all cases by these investigators.

SUMMARY

The reverse osmosis process is a very efficient means of producing a product water low in stable or radioactive inorganic and organic substances and free of microorganisms either in secondary-treated effluent, aqueous solutions or suspensions of multi-component solutions. Those substances which did permeate through the porous membrane in significant concentration were those species having a vapor pressure in the aqueous media appreciably greater than that of water.

A great potential for the reverse osmosis process lies in its use as a means of concentrating the substances in the waste water to where the waste water can be more efficiently treated.

APPENDIX

METHODS OF ANALYSIS

Standard methods (20) were used for the determination of all inorganic species, the surfactants -ABS and LAS-, the Coliform organisms for E. coli, and A. aerogenes. Gas chromatographic methods were used for the examination of the following: motor oil, lindane, BHC, DDT, TDE, p-chloro nitrobenzene, methyl formate, ethyl formate, ethyl acetate, methyl acetate, methyl propionate, ethyl propionate, the isopropyl ester of 2,4-D, acetone, phenol, chlorophenol, sodium acetate, sodium propionate, sodium valerate, and sodium isvalerate.

Spectrophotometric procedures were used to determine the following: tannic acid, humic acid, casein, peptone, starch, and cellulose hydrolyzates.

Thin-layer chromatographic methods were used in the analysis of L-glycine, L-lysine hydrochloride, DL-phenylalanine, and cellulose hydrolyzates.

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Table I. EFFECT OF SHRINKING ON MEMBRANE EFFICIENCY

Type	Original Pore Size m μ	Time of Immersion at 82°C min	Shrinking Percentage	Flux gals/ft ² /day	Reduction Factor
Schleicher & Schuell					
Type B-20	5	0	0	7.00	1.45
Type B-20	5	30	10	0.63	15.00
Type B-18	10-20	30	5.0-5.5	15-25	2.10-3.00
Type B-18	10-20	60	7.4-8.0	8-15	4.8-6.1
Type O-6	10-20	30	0	30-104	1.00-1.03
Type O-8	5	30	0	5	1.00
Gelman					
Type Gm-10	50	30	0	30	1.00

Conditions: Pressure 100 atm. feed of 1000 mg/l of Na ion in demineralized water.

Table II. Permeation of Single Inorganic Species
in Aqueous Solution.

Chemical Species	Flux ₂ gals/ft ² /day	Feed mg/l	pH	Product mg/l	pH	Reduction Percentage
Na ⁺ (NaCl)	14.8	10.700	-	1.500	-	86.5
	7.0	105.600	-	21.600	-	79.5
	5.5	1040.600	-	218.000	-	73.0
K ⁺ (KCl)	22.2	5.500	-	0.600	-	90.0
	17.6	53.400	-	13.5000	-	74.3
	15.5	521.000	-	316.000	-	39.4
NH ₄ ⁺ - N (NH ₄ Cl)	6.8	10.800	-	0.300	-	86.1
	5.2	48.800	-	1.400	-	86.9
	4.5	250.000	-	24.000	-	77.6
Ca ⁺² (CaCl ₂)	11.6	10.800	-	3.900	-	63.0
	11.0	48.800	-	20.000	-	59.0
	9.9	250.000	-	97.200	-	60.3
Mg ⁺² (MgCl ₂)	16.4	11.100	-	4.000	-	69.9
	14.2	54.000	-	21.700	-	60.0
	12.2	102.20	-	39.500	-	61.5
	11.0	266.800	-	102.800	-	61.5
Cd ⁺² (CdCl ₂)	25.8	0.097	-	0.030	-	69.0
	22.3	0.950	-	0.300	-	69.0
	20.8	9.250	-	2.790	-	70.0
Cu ⁺² (CuCl ₂)	14.6	0.960	5.50	0.040	5.61	95.9
	14.0	9.960	5.47	1.390	5.25	86.0
	13.2	100.50	5.20	17.800	5.20	82.3
Fe ⁺³ (FeCl ₃)	5.9	0.140	-	0.007	-	95.0
	14.1	1.090	-	0.074	-	93.2
	12.4	9.550	-	0.250	-	97.3
	9.2	93.600	-	1.98	-	97.9
Al ⁺³ (AlCl ₃)	11.8	1.260	4.82	0.017	5.31	98.7
	13.0	10.290	4.38	0.510	4.65	95.1
	11.2	150.100	4.08	5.600	3.90	94.7
Mn ⁺⁷ (K MnO ₄)	10.7	1.070	-	0.360	-	66.2
	8.7	10.800	-	3.650	-	66.2
	7.8	106.800	-	47.600	-	55.4

Table II. Continued

Chemical Species	Flux gals/ft ² /day	Feed mg/l	pH	Product mg/l	pH	Reduction Percentage
Cr ⁺⁶ (K ₂ CrO ₄)	12.3	0.078	-	0.001	-	99.6
	11.1	0.475	-	0.008	-	96.7
	10.5	5.010	-	0.250	-	95.0
	9.7	49.600	-	3.220	-	93.5
Cl ⁻ (NaCl)	18.1	12.100	-	1.100	-	90.9
	15.8	109.800	-	18.400	-	83.2
	14.4	1080.000	-	284.000	-	73.9
F ⁻ (NaF)	10.3	0.680	-	0.085	-	87.5
	10.8	6.040	-	0.900	-	85.1
	10.5	58.800	-	1.000	-	98.3
NO ₂ ⁻ - N (NaNO ₂)	9.2	0.438	5.60	0.075	6.10	82.9
	9.2	4.430	5.68	0.990	6.12	77.7
	8.3	40.100	-	11.250	-	72.0
NO ₃ ⁻ - N (NaNO ₃)	18.3	2.530	6.00	0.810	6.80	68.0
	18.3	25.600	6.35	8.110	6.27	68.3
	18.4	254.000	5.27	67.900	4.92	73.3
CN ⁻ (NaCN)	18.0	0.770	7.60	0.130	5.30	87.5
	17.8	7.550	9.00	1.105	6.50	85.1
	18.2	78.100	10.30	16.390	9.00	98.3
SO ₄ ⁻² (Na ₂ SO ₄)	14.9	13.800	5.75	< 1.0	6.1	> 93.0
	14.8	154.000	5.75	2.0	6.1	98.0
	14.8	1480.00	5.72	< 1.0	6.1	> 99.9
B ₄ O ₇ ⁻² - B Na ₂ B ₄ O ₇	16.2	1.310	8.70	0.820	5.90	37.0
	15.8	13.300	9.00	6.600	7.50	50.0
	15.9	132.00	9.10	64.000	8.30	51.0
SiO ₂ ⁻² (Na ₂ B ₄ O ₇)	22.0	1.800	8.9	0.415	8.9	77.0
	23.0	17.000	10.2	1.400	10.2	91.8
	23.3	175.000	11.3	11.460	11.3	93.5
PO ₄ ⁺³ (Na ₂ HPO ₄)	42.5	2.900	7.00	0.350	-	88.0
	40.6	29.700	7.50	3.550	-	88.0
	35.8	238.000	7.70	80.000	-	66.0

Table III. Permeation Data of an Ionic Mixture.

Ion	Feed		Product		Percentage Reduction	
	mg/l	m. moles/l	mg/l	m. moles/l	Mixture	Singly
Cl	108	3046	26.3	742	76	83
SO ₄	176	1832	7.5	78	96	98
PO ₄	32	337	2.5	26	92	88

Table IV. Permeation Data Trickling Filter Effluent

Parameter	Calculated as	Feed			Product		
		mg/l	meq/l	m.moles/l	mg/l	meq/l	m.moles/l
Ammonia	N	9.9	0.71	706	0.39	0.03	27
Hardness	CaCO ₃	124.0	--	1240	16.00	--	--
Calcium	Ca	21.1	1.06	526	1.60	0.08	40
Magnesium	Mg	17.3	1.42	728	2.90	0.24	120
Sodium	Na	48.0	2.08	2088	10.00	0.43	435
Potassium	K	9.4	<u>0.24</u>	<u>240</u>	0.40	<u>0.01</u>	<u>10</u>
Sum of Cations			5.51	4288		0.80	632
Total Alkalinity	CaCO ₃	164.0	3.28	1640	27.00	0.540	270
Chloride	Cl	21.5	0.61	606	4.00	0.110	113
Fluoride	F	0.6	0.03	31	0.02	0.001	1
Nitrate	N	8.2	0.59	587	3.84	0.270	274
Phosphate	PO ₄	20.4	0.64	215	0.54	0.017	6
Sulfate	SO ₄	21.0	<u>0.44</u>	<u>219</u>	2.00	<u>0.040</u>	<u>17</u>
Sum of Anions			5.59	3298		0.98	681
Conductance micromho at 25°C			535			85.7	
pH at 25°C			8.0			7.5	

Table V. Permeation Data Activated Sludge Effluent

Parameter	Calculated as	mg/l	Feed meq/l	m.moles/l	mg/l	Product meq/l	m.moles/l
Ammonia	N	18.20	1.30	1298	0.30	0.020	22.0
Hardness	CaCO ₃	125.0	--	1250	6.40	--	--
Calcium	Ca	24.0	1.20	600	<0.2	--	<10
Magnesium	Mg	15.6	1.30	515	1.49	0.12	62.0
Sodium	Na	53.0	2.30	2305	4.7	0.200	204.0
Potassium	K	11.20	0.29	286	0.6	0.170	17.0
Iron	Fe	0.09	<u>0.002</u>	<u>1.6</u>	0.01	<u>--</u>	<u>0.2</u>
Sum of Cations			6.39	6256		0.51	305.2
Total Alkalinity	CaCO ₃	161.0	3.22	1610	25	0.50	250
chlorides	Cl	23.0	0.65	649	2.5	0.070	71.0
Fluorides	F	1.07	0.06	56	0.07	0.004	4.0
Nitrates	N	<0.05	--	--	<0.05	--	--
Nitrite	N	0.0072	<0.01	0.5	0.0025	<0.001	0.2
Sulfate	SO ₄	19.0	0.40	198.0	1.9	0.040	20
Phosphate	PO ₄	14.5	0.46	153	1.0	0.032	11
Silica	SiO ₂	32.4	0.11	539	6.2	0.021	10
Polyphosphates	PO ₄	0.4	<u><0.01</u>	<u>4</u>	0.02	<u><0.01</u>	<u>0.02</u>
Sum of Anions			4.92	3205		0.66	366
pH	S.U.	7.5			7.2		

Table VI. Ionic Reduction Through a Membrane

Parameter	Calculated as	Percentage Reduction			
		Trickling Filter	Activated Sludge	Singly in aqueous solution	Singly based on total ionic strength
Ammonia	N	96	98	87	78
Hardness	CaCO ₃	87	94	--	--
Calcium	Ca	92	> 99	60	60
Magnesium	Mg	83	90	70	62
Sodium	Na	79	91	82	75
Potassium	K	96	95	88	60
Iron	Fe	--	87	95	97
Total Alkalinity	CaCO ₃	84	85	--	
Chloride	Cl	81	89	91	80
Fluoride	F	97	93	88	>98
Nitrate	N	53	--	68	75
Nitrite	N	--	65	83	72
Sulfate	SO ₄	90	90	93	99
Phosphate	PO ₄	97	93	88	66
Silica	SiO ₂	--	81	92	95
Polyphosphate	PO ₄	--	95	--	--

Table VII. Permeation of Chromium-51

Pressure Applied psig	Feed		Product		Percentage Reduction	
	Activity c/ml	pH	Activity c/ml	pH	Radioactive Species	Stable Species
1000	23,000	7.3	550	7.3	97.6	99.6
1500	23,000	7.3	250	7.3	98.9	

Table VIII. Permeation of Single Chemical Species
in Aqueous Solution or Dispersion

Chemical Species	Flux	Feed		Product		Reduction
	gals/ft ² /day	amt./l	pH	amt./l	pH	Percentage
Alkyl benzene	16	1.06 mg	--	0.06 mg	--	94.0
Sulfonate (ABS)	14	5.30 mg	--	0.52 mg	--	90.0
	18	10.60 mg	--	1.94 mg	--	82.0
Linear alkyl benzene	19	0.935 mg	7.2	0.120 mg	7.4	97.2
Sulfonate (LAS)	18	9.600 mg	5.0	0.095 mg	5.6	99.1
	18	10.100 mg	5.0	0.073 mg	5.8	99.3
Sodium oleate	7	38.800 mg	--	0.004 mg	--	99.9
Motor Oil, SAE-20	18	10.000 mg	--	<0.06 mg	--	>99.7
	19	10.000 mg	--	<0.06 mg	--	>99.7
Tannic acid	12	1.110 mg	4.7	0.100 mg	5.80	90.0
	11	11.100 mg	4.6	0.880 mg	7.30	92.1
	11	56.000 mg	3.9	2.880 mg	5.10	94.9
Lindane	18	50 ng	--	8.0 ng	--	84.0
	16	500 ng	--	133 ng	--	73.4
pp' - DDT	20	910 ng	--	<3 ng	--	>97.3
	19	5940 ng	--	<3 ng	--	>99.5
	17	7000 ng	--	14 ng	--	99.5

Table VIII. Continued

Chemical Species	Flux	Feed		Product		Reduction
	gals/ft ² /day	amt./l	pH	amt./l	pH	Percentage
Technical TDE (DDD)	15	23 μ g	--	<0.006 μ g	--	>99.9
	15	69 μ g	--	<0.006 μ g	--	>99.9
	14	532 μ g	--	<0.006 μ g	--	>99.9
Technical Benzene Hexachloride (BHC)	14	638 μ g	--	306 μ g	--	52
Casein	18	100 mg	6.6	<1.0 mg	6.4	>99
	17	1000 mg	7.8	1.6 mg	7.1	99
Peptone	17	100 mg	4.5	<2.0 mg	6.0	> 98
	17	1000 mg	4.0	<2.0 mg	6.1	99
Cellulose	24	8.4 mg	--	3.3 mg	--	61
	25	47.0 mg	--	14.6 mg	--	69
Soluble Starch	15	1.18 mg	--	<0.3 mg	--	>74
	16	8.30 mg	--	<0.3 mg	--	>91
	13	12.80 mg	--	<0.3 mg	--	>97
Technical	11	1000 units	6.5	<5 units	6.4	>99
Humic Acid	12	2000 units	6.5	<5 units	6.3	>99
	9	11000 units	9.2	<5 units	6.5	>99
	12	22000 units	9.9	<5 units	6.4	>99

Table VIII. Continued

Chemical Species	Flux gals/ft ² /day	Feed		Product		Reduction
		amt./l	pH	amt./l	pH	Percentage
p-Chloro nitrobenzene	9	95 µg	5.5	69 µg	5.9	27.3
	9	98 µg	5.3	720 µg	5.9	26.5
Methyl formate	10	80 mg	5.4	69 mg	5.4	14
Methyl acetate	9	93 mg	5.5	69 mg	5.2	26
Ethyl acetate	9	96 mg	6.0	54 mg	5.2	44
2,4-D Isopropyl ester	6	9.4 µg	--	< 0.02 µg	--	> 99.7
	5	59.6 µg	--	0.11 µg	--	99.8
	12	97.2 µg	--	4.30 µg	--	95.6
	9	673.0 µg	--	102.00 µg	--	84.9
Acetone	9	90 mg	5.3	69 mg	5.9	47

Table IX. Permeation of Chemical Species in a Multiple Component Aqueous Solution or Dispersion

Chemical Species	Flux gals/ft ² /day	Feed		Product		Reduction Percentage
		amt./l	pH	amt./l	pH	
phenol + chlorophenol	10.2		7.5		5.7	
phenol		10.1 mg		8.3 mg		17.8
chlorophenol		9.5 mg		3.2 mg		66.3
phenol + chlorophenol	11.6		8.7		7.0	
phenol		101.0 mg		82.8 mg		18.0
chlorophenol		106.0 mg		23 mg		78.3
phenol + chlorophenol	9.4		9.0		8.0	
phenol		1031 mg		844 mg		18.1
chlorophenol		1165 mg		267 mg		87.0
Ester mixture	9.6		4.2		4.4	
Methyl formate		98 mg		33 mg		66.6
Ethyl formate		87 mg		28 mg		67.8
Methyl propionate		92 mg		45 mg		51.0
Ethyl propionate		92 mg		40 mg		56.5
Amino Acid mixture	12					
L-glycine		10 mg	--	5 mg	--	50
L-lysine·HCl		10 mg	--	< 5 mg	--	> 95
DL-phenylalanine		10 mg	--	< 5 mg	--	> 95
Monocarboxylate mixture	19.4					
Sodium acetate		10.4 mg	--	1.70 mg	--	84

Table IX. Continued

Chemical Species	Flux	Feed		Product		Reduction
	gals/ft ² /day	amt./l	pH	amt./l	pH	Percentage
Sodium acetate		10.4 mg	--	1.70 mg	--	84
Sodium butyrate		9.5 mg	--	1.6 mg	--	80
Sodium valerate		9.2 mg	--	2.6 mg	--	72
Sodium isovalerate		9.2 mg	--	2.6 mg	--	72
Monocarboxylate mixture	14.5					
Sodium acetate		104.2 mg	--	24.2 mg	--	77.3
Sodium propionate		99.1 mg	--	26.2 mg	--	74.2
Sodium butyrate		95.1 mg	--	26.5 mg	--	72.6
Sodium valerate		92.0 mg	--	30.0 mg	--	69.4
Sodium isovalerate		93.0 mg	--	28.1 mg	--	70.6
Monocarboxylate mixture	8.2					
Sodium acetate		1042.0 mg	--	324.0 mg	--	64.9
Sodium propionate		991.0 mg	--	356.0 mg	--	64.2
Sodium butyrate		951.0 mg	--	323.0 mg	--	65.2
Sodium valerate		920.0 mg	--	293.0 mg	--	68.1
Sodium isovalerate		930.0 mg	--	320.0 mg	--	65.0
Mixture	8.2					
2,4-D isopropyl ester		65.8 μ g	--	30.0 μ g	--	54.4
p-chloro nitrobenzene		82.0 μ g	--	61.0 μ g	--	25.6
phenol		10.3 mg	--	8.8 mg	--	13.8
chlorophenol		9.8 mg	--	6.2 mg	--	36.7
Acetone		100.7 mg	--	67.1 mg	--	33.3
Ethylacetate		100 mg	--	39.7 mg	--	60.4

Table X. Permeation of Chemical Species in Treated
Waste Water Effluents

Chemical Species	Flux	Feed		Product		Reduction
	gals/ft ² /day	amt./l	pH	amt./l	pH	Percentage
Alkyl benzene	16 ¹	3.24 mg	--	0.24 mg	--	93.0
Sulfonate (ABS)						
Carbohydrate	12 ¹	13.5 mg	7.4	6.0	8.1	56
(Anthrone reactive)	17 ²	4.30 mg	7.8	0.3	6.2	93
Mixture	9 ²					
2,4-D isopropyl ester		87.2 µg	--	5.13 µg	--	94.1
p-chloro nitrobenzene		118.7 µg	--	70.83 µg	--	40.4
phenol		9.5 mg	--	7.4 mg	--	21.4
chlorophenol		9.6 mg	--	4.8 mg	--	50
DDT		97.5 µg	--	<2.0 µg	--	>98
Ethyl acetate		72.6 µg	--	24.7 µg	--	66.0
Acetone		95.8 µg	--	58.3 µg	--	39.1

¹ - Trickling filter effluent

² - Activated sludge effluent

Table XI - Comparison of the Per Cent Reduction of Specific Species in Effluents, Mixtures, and Single-Solute Systems.

Chemical Species	Percentage Reduction Specific Species in		
	Effluent	Mixture	Singly
Alkyl benzene Sulfonates (ABS)	93.0 ¹	--	90.0
Carbohydrate			
Anthrone reactive material	56 ¹	--	--
	93 ²	--	--
Cellulose	--		61
Starch	--		97
Mixture			
2,40D isopropyl ester	94.1 ²	54.4	95.6
P-chloro nitrobenzene	40.4	25.6	26.5
phenol	21.4	13.8	17.8*
chlorophenol	50.0	36.7	66.3*
DDT	98	--	97.3
Ethyl Acetate	66.0	60.4	44.0
Acetone	39.1	33/3	47.0
Amino Acids			
L-glycine	82 ²	50	--
DL-phenylalanine	82	95	--
L-lysine hydrochloride	90	95	--

* phenol-chlorophenol mixture, ¹-trickling filter effluent, ²-activated sludge treated effluent

Table XII. Permeation of Microorganism Through a Cellulose Acetate Membrane.

Type	Feed organisms/ml	Product organisms/ml	Raffinate organisms/ml
Coliform	MPN 89.5/ml	Absent	MPN 80/ml
	MPN 895/ml	Absent	MPN 905/ml
	MPN 8950/ml	Absent	MPN 7500/ml
<u>S. narcesence</u>	105/ml	Absent	105/ml
	1050/ml	Absent	1050/ml
	10500/ml	Absent	10500/ml
<u>E. coli</u>	504/ml	Present	---
<u>E. coli</u>	495/ml	Absent	---
<u>A. aerogenes</u>	160,000	Absent	---
Coliphage T-7	Growth	Absent	Growth
Coliphage ϕ X-175	Growth	Absent	Growth